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Quarterly Progress Report #1

For the period February 17, 1964 to May 17, 1964

on

FACTORS CONTROLLING THE STRENGTH OF COMPOSITE BODIES (INTERPHASE FRACTURING OF COMPOSITE BODIES)

for

BUREAU OF NAVAL WEAPONS
Washington 25, D. C.

by

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Since this is a progress report, the information herein is tentative and subject to change, correction and modification.

ABSTRACT

The factors that control the fracture toughness of adhesive joints at ambient temperature and humidity are the adherend material, its surface finish and geometry, the adhesive composition and curing procedure, the bond thickness, the loading rate and resulting crack velocity and fracture type. Of major concern is the fracture type and the range of crack velocities associated with each type. Because toughness is dependent on propagation rate, it is necessary to measure the parameters of fast moving cracks. Consequently, the major effort during this first quarterly period was directed toward assembling and evaluating high strain rate recording equipment. It is now possible by means of an oscilloscope to record load-deflection, deflection-time, load-time and crack length-time simultaneously up to and during crack propagation. In addition, the fracture surface can be marked with a ripple pattern of known frequency so that the cracking rate can be measured over microscopic distances.

Preliminary tests indicate that a stoichiometric composition of Dow 332 and TEPA results in joints with a toughness ($\mathcal{G}_{Ic}$) of 1/4 to 1/2 that of Budd Photostress Type A which was used previously. The crack extension force for a running crack is 75 percent that of a stationary crack, and aluminum bond surface finished of 10 and 25 micro-inch (rms) produce identical toughness.

INTRODUCTION

Previous work on the application of fracture mechanics to adhesive joints indicated that the toughness of the system was controlled by the rate at which the crack propagated through the adhesive. This rate generally was not a reflection of the speed at which cracking energy was supplied to the sample, but appeared to depend on the fracture type: abrupt jumping cracks have smooth, bright fracture surfaces and resulted in low toughnesses, while slow moving cracks produced dull surfaces with high toughnesses. Bond thickness appeared to affect toughness by encouraging either a bright or dull fracture. At moderate displacement rates, the bond thickness effect diminished and fracturing occurred more and more by a series of jumps.

Since crack velocities during the jump outran standard pen recording speeds, higher rate recording equipment was necessary to evaluate the toughness of running cracks. Consequently, an oscilloscope which plots the load and extension variables has been added to the standard testing procedure previously described (1). To macroscopically measure cracking rates, conducting paint lines are placed at quarter inch intervals along the sample length and, as these break, they locate the crack front. To measure rates within a single jump, a much finer measuring technique was required. This is accomplished by supplying a known audio signal to a bell coil that is mounted onto one half the test sample and made to oscillate by surrounding it with a powerful magnet. The vibrations cause the fracture to deflect slightly from its path leaving a ripple pattern whose period is measured after the test is completed to obtain a crack velocity profile during propagation.

The epoxy has been changed from Budd Photostress Type A to the more generally applicable Dow 332 resin and TEPA hardener. Since this same system is being used by the three cooperating contractors on the study of composite bodies, data collected by each contractor will be interchangeable.

In the previous study of a glass-epoxy-glass system, changes in bond surface roughness were found to produce a two-fold variation in toughness. Consequently, some preliminary data is also being collected on the effect of surface finish of aluminum and steel adherends.

MATERIALS AND PROCEDURES

Epoxy Formulation

In the first attempts to measure toughness of joints it appeared necessary to determine the stress field surrounding the crack tip by photoelastic means. Consequently, Budd Photostress Type A was selected as the adhesive. Since $\mathcal{G}_{Ic}$ is now being determined by energy methods, a very pure epoxy resin and hardener system, i.e. Dow 332 and TEPA, has been substituted so that the three contractors in the program will be readily able to compare appropriate experimental results.

Sample Preparation

Adherend bond surfaces were prepared in a manner similar to that described in Ref. 2. Cleaning solutions and procedures were of a standard type for epoxy joints (see, for example, Ref. 3). Changes made in the course of this testing program include the use of a milled adherend bond surface in addition to ground ones to obtain a variety of surface finishes as well as the use of glass cast silastic tapes to obtain uniform thin coatings of epoxy (5 to 15 mil max) on the sides of the sample. These flat, insulated side surfaces were necessary for putting on painted circuit lines as discussed later. Homogeneous epoxy reference samples for tensile and fracture toughness evaluation were also made using silastic, cast against glass forms as a mold.

Modified Tensile Mode Toughness ($\mathscr{G}_{Ic}$) Specimen

Samples used previously had the loading hole placed on the crack line. The loading holes for this study were moved to the neutral axis of the two adherends to minimize the tendency for rotation and twisting of the adherends during testing. The stiffer loading grips that this hole design permitted enables evaluation of $\mathscr{G}_{Ic}$ using an Instron tensile machine, which is a good deal softer than the device used initially. A schematic drawing of a sample ready for testing is shown in Fig. 1. A recheck of compliance-crack length data, using calibration bars, was in good agreement with the equations used in developing the nomograph (1).

Measurement of Crack Rate

The influence of cracking rate on fracture toughness of adhesive joints is probably of the same order of importance as stress state. Since conventional pen recording equipment cannot follow even moderately fast loading times to fracture, i.e. less than 1/2 to 1/10 sec., other techniques had to be used to follow crack propagation and establish the influence of crack velocity on toughness.

A four-channel x-y Tektronix storage oscilloscope has been adapted to simultaneously record load (from an in-series load cell) vs. deflection, deflection vs. time, load vs. time and crack front position vs. time. The latter is obtained by painting lines of conductive paint across the expected fracture path at quarter inch intervals as shown in Fig. 2. In series with each of these lines is a resistor and these are all connected in parallel. The parallel bank is in series with an external resistive element, R_M across which the voltage is measured. As the crack passes each conductive line, it removes one resistor from the parallel circuit so that the measured voltage E_{Ma} decreases. This decrease is proportional to the number of lines broken, and so long as R_M is small compared with the individual resistances, the crack length is given by:

$$E_{Mo} - E_{Ma} = (N_o - N_a)\frac{E_1}{R_1} R_M$$

where

E_{Mo} = 1.67 v. = initial voltage across R_M

E_{Ma} = voltage across R_M as circuit lines are broken

N_o = 40 lines spaced 1/4 inch apart = no. of original elements

N_a = no. of unbroken elements

R_1 = 20,000 ohms

R_M = 100 ohms

A schematic diagram of the test set-up is shown in Fig. 3, and a block diagram of the circuitry in Fig. 4. A diagram of the typical data obtained on a single sample is shown in Fig. 5. At slow head speeds the oscilloscope data is recorded simultaneously with that of the Instron and an x-y recorder driven by separate load and deflection transducers: good correlation between all instruments has been obtained.

Crack velocity on a fine scale is obtained by ripple markings on the fractured surfaces at the completion of the test. The markings are made by vibrating one half of the sample while the crack is running. This is accomplished by an oscillator consisting of an audio generator coupled to a bell coil through a 60 watt audio amplifier. The bell coil transducer is bolted to one half of the specimen and magnetic force supplied via a 5200 Gauss magnet. The oscillating frequencies used to date have been 50 and 100 cps. Ripple marking separated by from .1 to .25 inch at 100 cps. show the crack velocity to vary from 1.7 to 4 ft./sec. This ripple-marking crack rate measuring procedure was suggested by NRL.

TEST RESULTS

Only a very limited amount of test data has been collected to date. Hence, all of the test results listed below must be considered as tentative. Nevertheless, the fracture toughness of a stoichiometric composition of Dow 332 resin with TEPA (14.2 parts TEPA to 100 parts resin) has a toughness one quarter to one half of that found for the previously used Budd epoxy. An examination of the scope trace of the load displacement curve indicates that the fracture toughness of a running crack is only about 25% lower than that of a starting crack.

Crack velocities, $\dot{a}$'s, observed to date, i.e. 1.7 to 4 ft./sec., are much lower than the 100 to 500 ft./sec. cracks expected in this material at the lowest point in the $\mathcal{G}_{Ic}$ vs. $\dot{a}$ curve. Hence, it is expected that $\mathcal{G}_{Ic}$ will continue to decrease with increasing cracking rates over two more orders of magnitude. The ripple markings indicate that as the crack jumps from one stop to the next, $\dot{a}$ attains its maximum value very soon after motion is initiated and then decreases over a relatively long time period.

The effect of bond surface finish seems to be insignificant for the aluminum-epoxy-aluminum system, although surfaces of only ten and twenty-five micro-inches have been evaluated to date.

FUTURE WORK

With the ability to measure crack velocity on both a micro- and a macroscopic basis, the influence of strain rate on fracture toughness will now be determined. This data is expected to supplement the experiments now under way at Alpha R and D and the University of Illinois. Although the effect of combining $\mathcal{G}_{Ic}$ and $\mathcal{G}_{IIc}$ measurements have not been attempted as yet, an apparatus has been designed for this type of testing, and it is expected that these data will assist in understanding the bulk properties being measured at the University of Illinois.

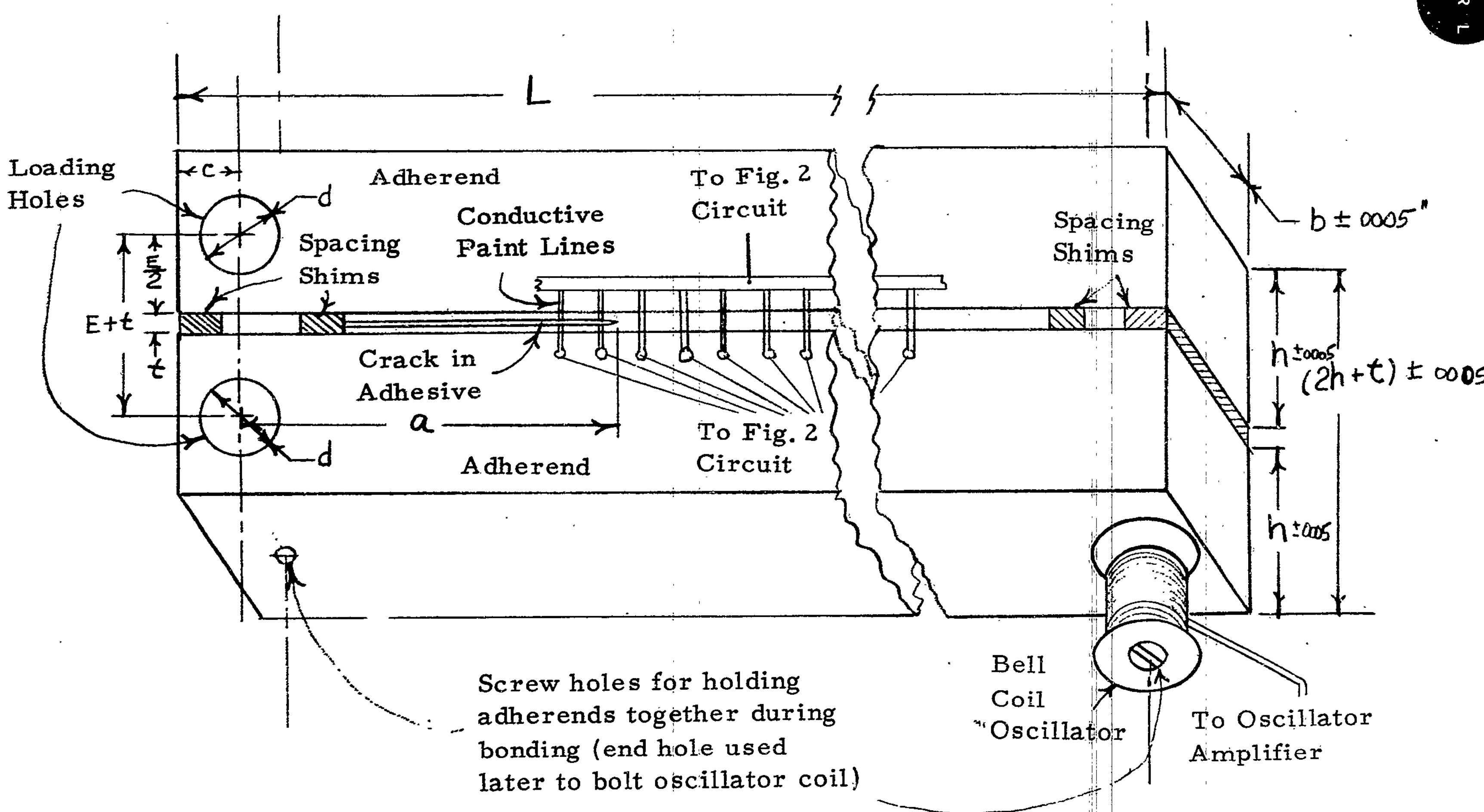

Fig. 1 MODIFIED TENSILE MODE TOUGHNESS, $\mathcal{G}_{Ic}$, SPECIMEN, SHOWING NEW POSITION OF LOADING HOLES, BELL COIL OSCILLATOR AND PRINTED CIRCUIT LINES. (FOR THE INITIAL STUDY h = 1", L = 12", b = 1/2", d = 1/2", c = 3/8", E = 1".)

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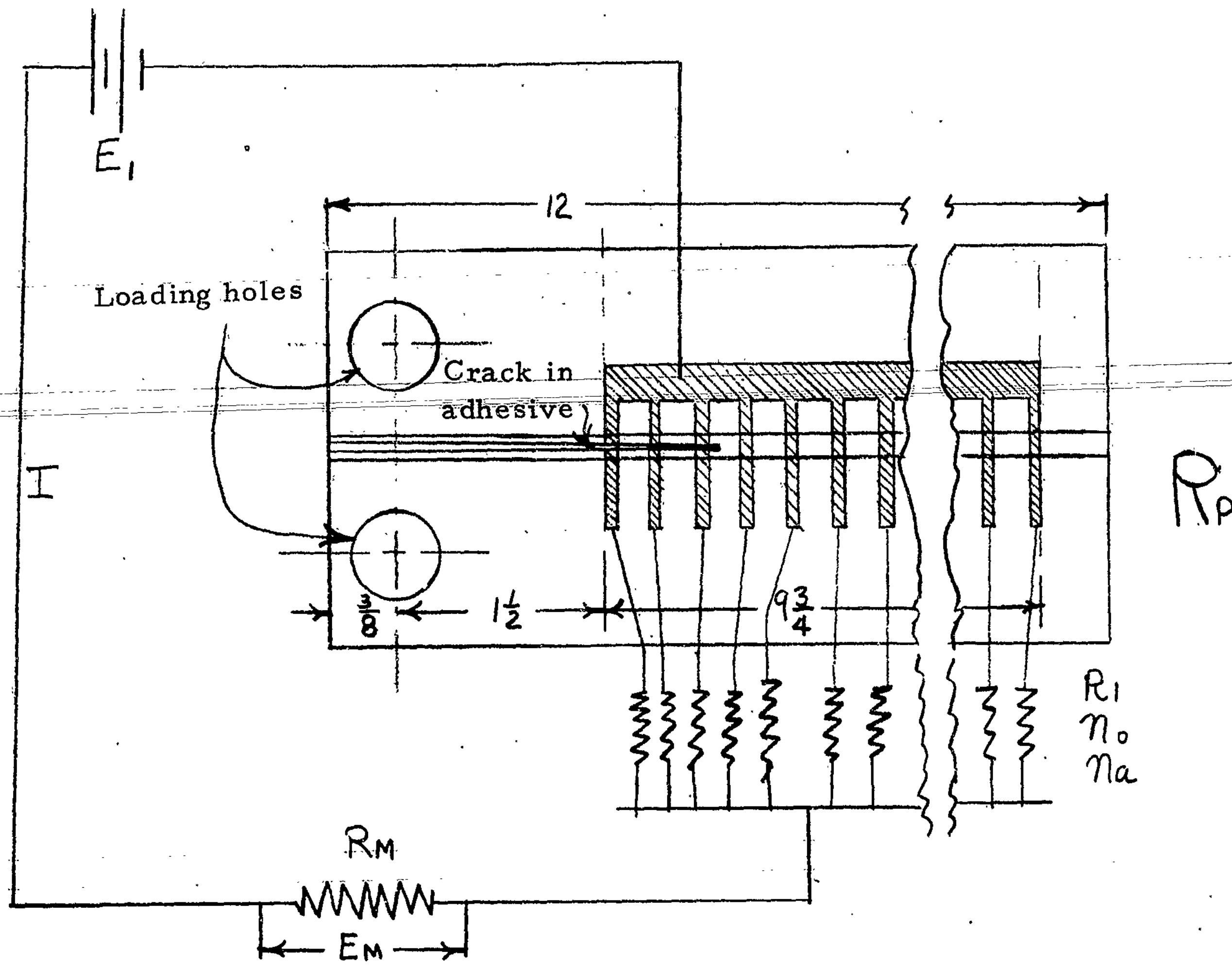

Fig. 2 PAINTED CIRCUIT LINE SCHEMATIC DIAGRAM.

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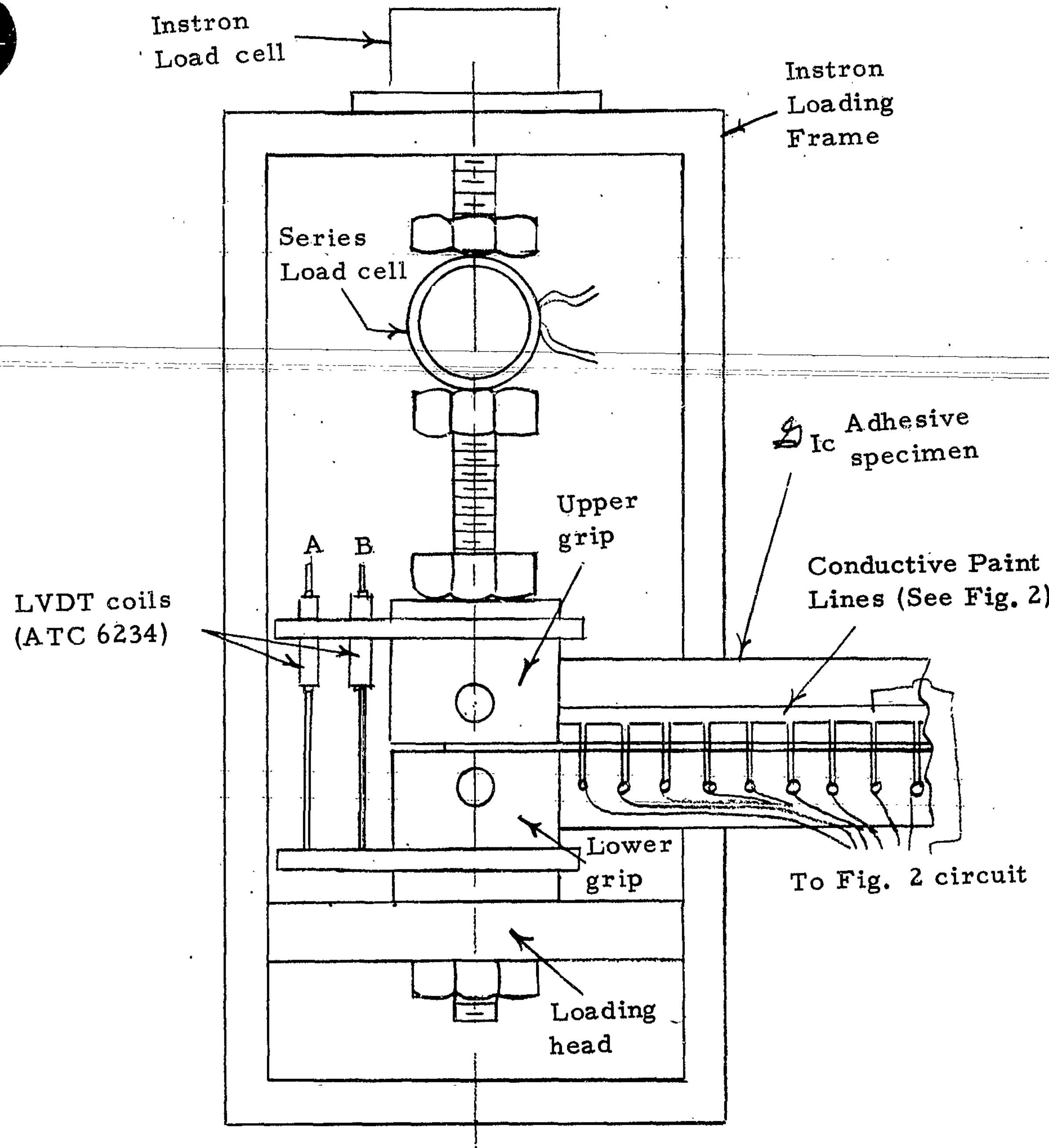

Fig. 3 INSTRUMENTAL MEASURING AND LOADING ARRANGEMENT USING INSTRON TENSILE MACHINE (SCHEMATIC).

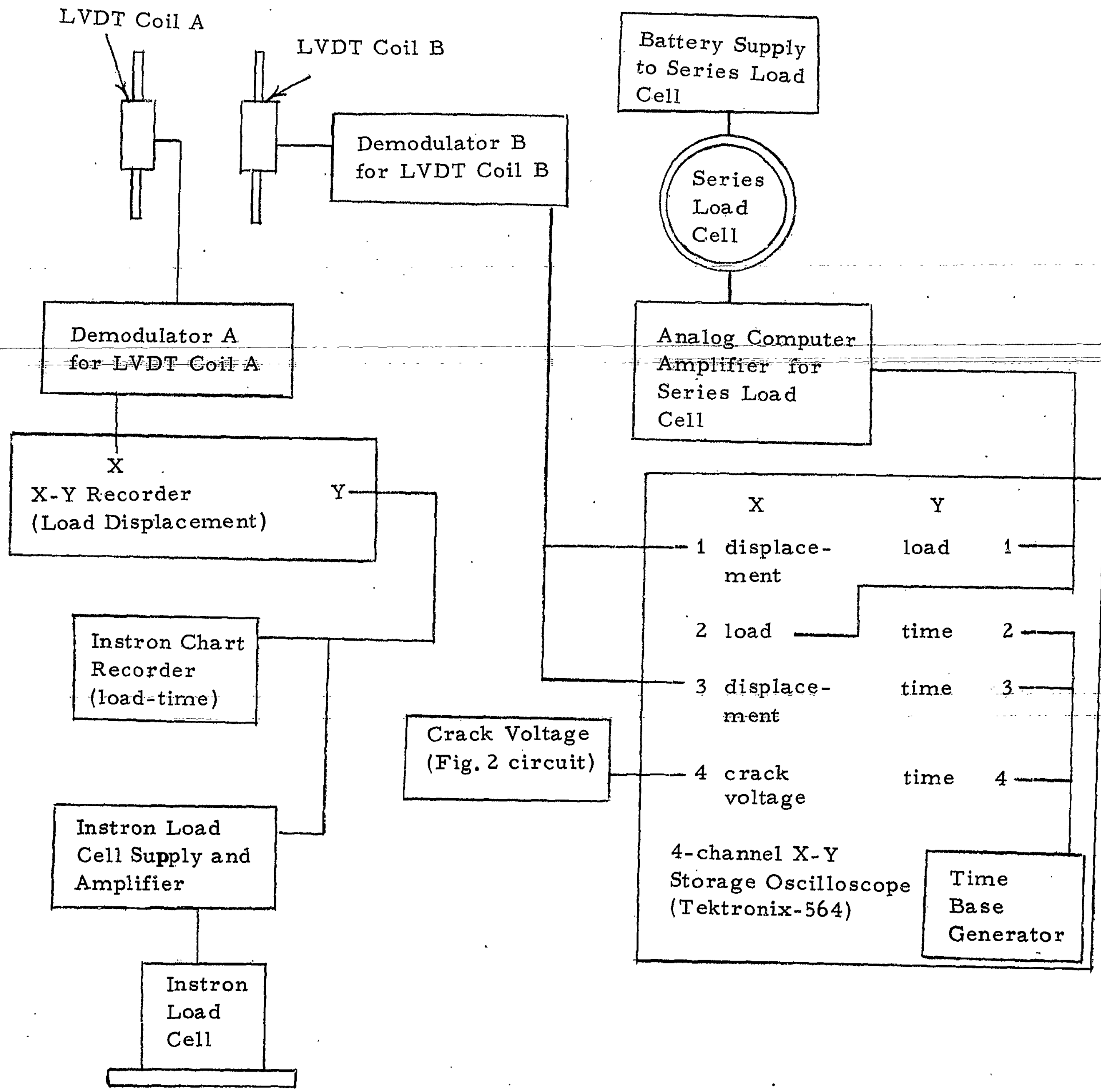

Fig. 4 BLOCK DIAGRAM OF LOW AND HIGH STRAIN RATE MEASURING INSTRUMENTS FOR δ_{Ic} DETERMINATION

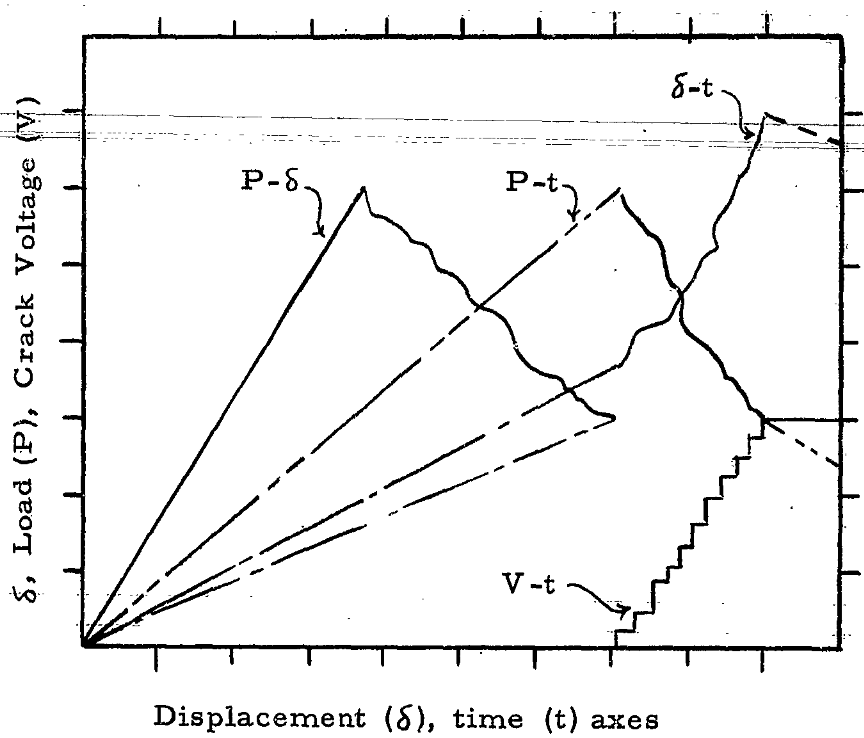

Fig. 5 Identification of 4 Channel Oscilloscope Traces - Slow Moving Crack

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